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X-RAY INVESTIGATION OF BALLISTIC YARNS.(U)
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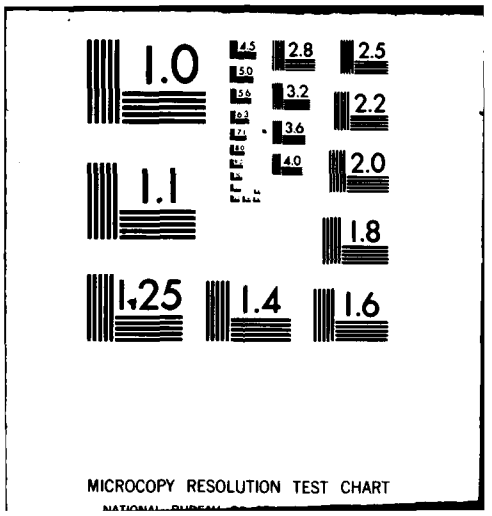
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X-RAY INVESTIGATION OF BALLISTIC YARNS

C. RICHARD DESPER
POLYMER RESEARCH DIVISION

December 1979

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ABSTRACT

A series of ballistic polyamide yarns, comprising 12 Kevlar samples and one Nylon 66 sample, were examined by X-ray diffraction. The nine Kevlar 29 and three Kevlar 49 yarns were identified by their lattice parameters as poly(p-phenyleneterephthalamide) rather than poly(p-benzamide). The normalized equatorial scans show a somewhat greater crystallite size or perfection for the Kevlar 49 samples compared to the Kevlar 29 samples. The equatorial scan of the Nylon 66 sample yielded a Crystal Perfection Index of 72 percent. Orientation functions in the range 0.95 to 0.97 were measured for the Kevlar samples, while the corresponding value for the Nylon 66 sample was 0.881.

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INTRODUCTION

A total of thirteen ballistic yarns submitted by the U.S. Army Natick Research and Development Command have been examined by X-ray diffraction. Of these, twelve are of the Kevlar-type aromatic polyamide, and one is a ballistic grade of nylon 66. Identification numbers were assigned to distinguish the samples, and these, along with the descriptions received with the samples, are listed in Table 1. The twelve Kevlar samples consist of four basic yarn types. For each type, three samples are provided: a control, a yarn subjected to tensile rupture, and a yarn exposed to UV radiation for 40 hours and then subjected to tensile rupture. The nylon 66 sample was a control, subjected to neither UV radiation nor tensile rupture.

The yarns were mounted in the Picker FACS-1 X-ray diffraction unit, using a pinhole-collimated primary beam of 0.5-mm diameter. A single multifilament yarn was placed in the beam, making every effort to align the filaments parallel to each other. Since the experiment requires a certain minimum amount of sample in the beam, no attempt was made to study individual filaments or to study the ruptured yarns at the point of the break. Thus, the ruptured yarns represent material subjected to breaking stress and unloaded.

The intensity profiles were measured as a function of either Bragg angle (2θ) or orientation angle (χ). The instrument resolution was estimated as 0.3° for 2θ and 2° for χ. Data was collected by counting techniques under computer control as previously described,[1] using monochromatic radiation at 1.5418 A wavelength. The data tapes were processed off-line using a Hewlett-Packard 9830 calculator with XY plotter.

Table 1. SAMPLE DESCRIPTION

Material	Sample	Code*	Type	Tex, g/10,000 m
Kevlar 29	29-1	C	964	113.6
	29-2	R	964	113.6
	29-3	UVR	964	113.6
	29-4	C	964	170.6
	29-5	R	964	170.6
	29-6	UVR	964	170.6
	29-7	C	950	174.3
	29-8	R	950	174.3
	29-9	UVR	950	174.3
Kevlar 49	49-1	C	N/A	163.0
	49-2	R	N/A	163.0
	49-3	UVR	N/A	163.0
Nylon 66	66-1	C	N/A	116.7

*C-control, R-ruptured, UVR-exposed to UV and ruptured.

1. DESPER, C. R. *A Computer-Controlled X-Ray Diffractometer for Texture Studies of Polycrystalline Materials.* Adv. X-Ray Anal., v. 12, 1969, p. 404.

CRYSTALLOGRAPHIC RESULTS - KEVLAR YARNS

Equatorial scans for the Kevlar samples in the 2Θ direction are shown in Figure 1. The two strong reflections are identified as the (110) and (200) reflections of poly(p-phenyleneterephthalamide) at 21.50° and 23.85°. The (004) reflection was observed in meridional scans (not shown). The observed spacings and lattice parameters are given in Table 2. There were not detectable differences in these spacings within the nine Kevlar 29 and the three Kevlar 49 samples. However, small differences, close to the error limit, were noted between the Kevlar 29 and the Kevlar 49 samples. These differences, if they are real, could result from different processing conditions.

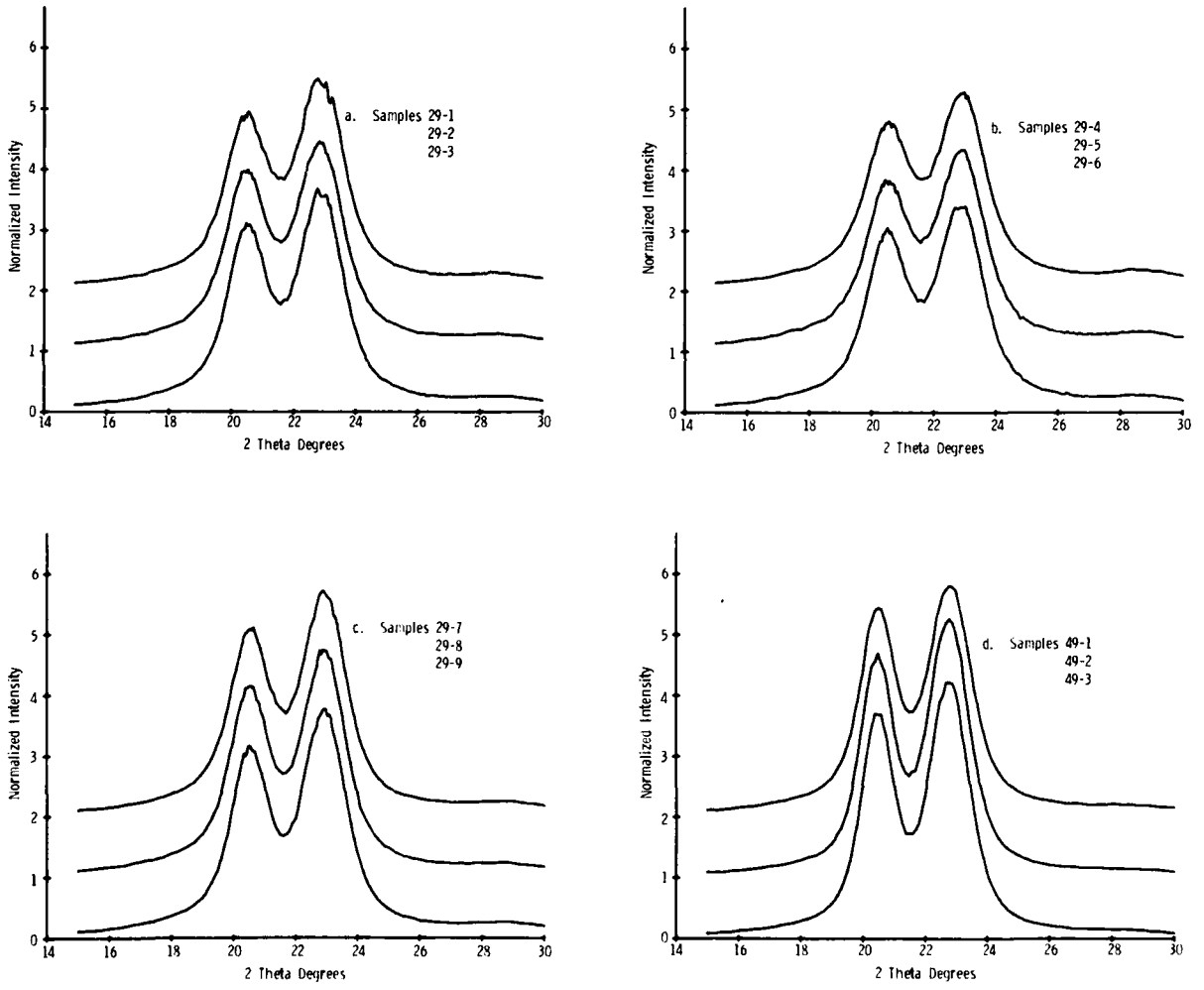

Figure 1. Normalized equatorial X-ray diffraction scans, using λ = 1.5418 Å. Bottom curve, no offset. Center curve, offset 1 unit upward. Top curve, offset 2 units upward.

There has been confusion at times as to the exact chemical nature of the various aromatic polyamide yarns. Tashiro, Kobayashi, and Tadokoro[2] identified "Kevlar" as poly(p-phenyleneterephthalamide) and "PRD-49, Type I" as poly(p-benzamide). The two fibers bear a close similarity in chemical structure and in diffraction spacings. Comparison of the experimental lattice parameters in Table 2 with the literature values in Table 3 indicates that the present Kevlar samples are all poly(p-phenyleneterephthalamide) in agreement with Tashiro and co-workers.[2]

Table 2. KEVLAR CRYSTAL SPACINGS AND UNIT CELL PARAMETERS (in Angstroms)

Parameters	Samples 29-1 Through 29-9	49-1 Through 49-3	Precision
d(110)	4.332	4.332	0.010
d(200)	3.892	3.900	0.010
d(004)	3.209	3.224	0.010
a	7.784	7.800	0.020
b	5.214	5.209	0.020
c	12.84	12.90	0.040

Table 3. LITERATURE VALUES[2] FOR CRYSTAL LATTICE PARAMETERS (in Angstroms)

Poly(p-phenyleneterephthalamide)	a = 7.80
	b = 5.19
	c = 12.9
Poly(p-benzamide)	a = 7.71
	b = 5.14
	c = 12.8

DIFFRACTION PROFILES - KEVLAR SAMPLES

The raw intensity curves will vary in level since the mass in the X-ray beam varies from sample to sample. To put the data from different samples on a common basis, a normalization procedure was used: each intensity curve, after correction for instrument background, was divided by the integrated average intensity in the range $2\theta = 15°$ to $30°$. With this procedure, several diffraction curves may be compared on the same graph, each curve offset from its neighbor by one unit of integrated intensity. Visual comparison in Figure 1a shows that there is little difference in the equatorial diffraction curves of samples 29-1, 29-2, and 29.3. These three samples are the control, the tensile ruptured, and the UV exposed and tensile ruptured specimens of a single yarn type and tex. At a qualitative level, the diffraction curve of the yarn has changed very little. The same conclusion may be drawn for the other Kevlar yarn types.

Examining Figure 1, it was evident that there is a marked difference between the curves for the nine Kevlar 29 samples and those of the three Kevlar 49 samples. To demonstrate this effect, the intensity data for the Kevlar 29 and for the Kevlar 49 samples were mathematically summed to yield the curve labeled "29-SUM" and "49-SUM" in Figure 2. The differences between the two curves is quite apparent. Although the peak positions are essentially unchanged, the peaks in the Kevlar 49 pattern are sharper, and the minimum between the peaks is lower. The sharper peaks indicate that the crystallite size and/or perfection is higher for the Kevlar 49 samples, probably reflecting a difference in processing conditions.

To place the differences in peak sharpness on a quantitative basis, a procedure was devised to calculate what shall be called an "integral half-breadth." The mathematical basis is illustrated in Figure 3. A baseline is drawn between

2. TASHIRO, K., KOBAYASHI, M., and TADOKORO, H. *Elastic Moduli and Molecular Structures of Several Crystalline Polymers, Including Aromatic Polyamides.* Macromolecules, v. 10, 1977, p. 413.

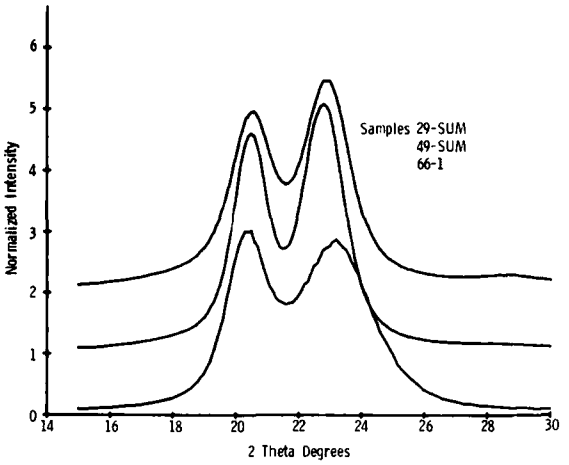

Figure 2. Comparison of averaged equatorial scans for nine Kevlar 29, three Kevlar 49, and Nylon 66 samples.

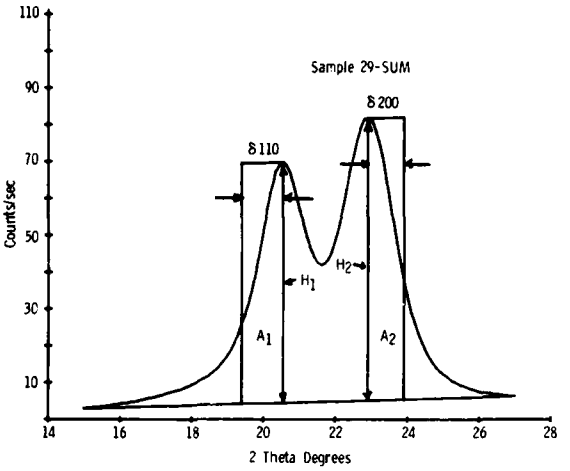

Figure 3. Method of determining the integral half-breadths δ(110) and δ(200) for Kevlar samples.

the data points at $2\Theta = 15°$ and 27°, and vertical lines are dropped from the two peak positions to the baseline. The calculated area between the curve and the baseline to the left of the (110) peak is designated A_1, while the analogous area to the right of the (200) peak is designated A_2. The two peak intensities above baseline are designated H_1 and H_2. We may now define the integral half-breadth as follows:

$$\delta(110) = A_1/H_1, \text{ and}$$

$$\delta(200) = A_2/H_2.$$

In essence, the integral half-breadth δ(110) is defined as the width of a parallelogram of height H_1 possessing the same area A_1 as the diffraction profile from 15° to the (110) peak position. Likewise, δ(200) is defined to yield a parallelogram of area A_2.

The values of δ(110) and δ(200) determined for the Kevlar samples are listed in Table 4. As a rule, the variation of a given integral half-breadth is small within the various specimens of the Kevlar 29 or of the Kevlar 49 type. However, the integral half-breadth values for the 29-SUM data are both about 0.2° greater than the corresponding values for 49-SUM, a sizeable difference. This confirms the conclusion that the crystallites have greater perfection or size in Kevlar 49 than in Kevlar 29.

The integral half-breadths are also affected by tensile rupture and UV exposure, although the effects are smaller than that of fiber type. Inspection of Table 4 indicates that δ(110), in particular, is reduced for the samples exposed to ultraviolet radiation and rupture. To focus in on this effect, the data for the control, rupture, and UV plus rupture samples of Kevlar 29 were summed to give the lumped effects of these treatments. These data, listed under the averaged scans, show a reduction of 0.06° in δ(110) with UV and rupture. The

effect on δ(200) for Kevlar 29 is in the same direction but smaller. The limited Kevlar 49 data shows effects in the same direction. The overall data indicate a definite, albeit small, reduction in integral half-breadth with UV exposure and rupture, and less of an effect with rupture alone. This is interpreted as a reduction in crystal defect content, leading to an improvement in the crystalline order. It is possible that the crystal defects were amorphous regions which are, to some extent, more susceptible to the effects of UV exposure and/or tensile rupture.

CRYSTAL PERFECTION IN NYLON 66

The nylon 66 sample shows two strong equatorial diffraction peaks (see Figure 2) in the 2θ = 15 to 30° range. These are identified as the (100) reflection and the unresolved (010) - (110) pair of the triclinic "alpha" structure.[3] The latter peak is notably broader than the former, probably because of its compound nature. The peak positions correspond to d spacings of 4.358 and 3.837 Å, of which the latter is notably larger than the value for the (100) spacing for the structure of Bunn and Garner,[3] as shown in Table 5. Dismore and Statton[4]

Table 4. KEVLAR FIBER EQUATORIAL SCANS; INTEGRAL HALF-BREADTHS

Sample	Code	δ(110), Degrees	δ(200), Degrees
29-1	C	1.13	1.04
29-2	R	1.15	1.02
29-3	UVR	1.06	1.03
29-4	C	1.16	1.08
29-5	R	1.17	1.04
29-6	UVR	1.10	1.05
29-7	C	1.07	1.03
29-8	R	1.08	1.01
29-9	UVR	1.02	1.02
49-1	C	0.94	0.91
49-2	R	0.89	0.83
49-3	UVR	0.89	0.83
29-SUM	ALL	1.12	1.04
49-SUM	ALL	0.91	0.86
29-C	C	1.12	1.05
29-R	R	1.13	1.02
29-UVR	UVR	1.06	1.03

Table 5. CRYSTALLOGRAPHIC DATA FOR NYLON SAMPLE 66-1

	hk0	2θ, Degrees	d, Angstroms
Present Work:	(100)	20.38	4.358
	(010)+(110)	23.18	3.837
"Perfect Crystal" Values:[3]	(100)	20.37	4.36
	(010)	24.09	3.69
	(110)	24.43	3.64

Crystal Perfection Index:[4] 72%

3. BUNN, C. W., and GARNER, E. V. *The Crystal Structures of Two Polyamides ('Nylons').* Proc. Royal Soc., (London), v. A-189, 1947, p. 39.
4. DISMORE, P. F., and STATTON, W. O. *Chain Folding in Oriented Nylon 66 Fibers.* J. Polymer Science: Part C, no. 13, 1966, p. 133-148.

attributed the increase in this spacing to crystal imperfection, and proposed an empirical crystal perfection index (CPI) based on the ratio of the two observed spacings. The calculated crystal perfection index for sample 66-1 is 72%, which is rather low for a nylon 66. Care must be exercised in interpreting this figure. Although the CPI may correlate with density, it is not a weight fraction crystallinity; indeed the concept of weight fraction crystallinity is probably erroneous for nylon fibers. Rather, it is a number which tells something about the extent of perfection of the fiber structure, particularly in terms of perfection in the formation of hydrogen-bonded sheets, which lie parallel to the (010) crystal planes.

A more complete characterization of lateral order in nylon 66 would require that additional factors such as crystallite size and the possible presence of a second crystal phase, the "gamma" phase, be taken into account. Such a multiple parameter problem may be solved only through mathematical resolution of the diffraction curve into its component peaks. Heuvel, Huisman, and Lynd[5,6] have proposed an unambiguous curve resolution procedure for nylon 6, which presents a similar problem. Their approach involves considering variations in peak positions, breadths, and line shapes to achieve the best fit of the data. From such an analysis information regarding crystallite size and gamma phase content, as well as crystal perfection, are extracted. Such a degree of sophistication is not, however, appropriate in the present context.

CRYSTALLINE ORIENTATION - KEVLAR SAMPLES

The crystalline orientation distributions of the (110) and (200) planes of the Kevlar samples were measured as a function of orientation angle χ at 1° intervals, holding 2θ fixed at the two peak values. The baseline intensities at these 2θ values, interpolated from intensity values at 2θ = 15° and 30°, were subtracted from the χ scan intensities to eliminate noncrystalline scattering. Then the orientation functions f(110) and f(200) were computed using established methods.[7] The chain direction orientation functions f(c) were then calculated from f(110) and f(200) using:

$$f(c) = [(1-2\sin^2\rho(110,a))f(200)-f(110)]/\sin^2\rho(110,a)$$

where $\rho(110,a) = 56.3°$ is the angle in the unit cell between the a axis and the (110) normal. The equation was derived for the orthorhombic system of polyethylene and is applicable in the pseudo-orthorhombic Kevlar case. The half-intensity orientation angles $\chi(1/2)$ were also calculated from the corrected intensity curve. (This quantity is defined[7] as the angular range in which the intensity drops from maximum to half-maximum intensity.) The orientation distributions for the Kevlar samples approximate a Gaussian shape as shown in Figure 4a, given by:

5. HEUVEL, H. M., HUISMAN, R., and LIND, K. C. J. B. *Quantitative Information from X-Ray Diffraction of Nylon-6 Yarns. I. Development of a Model for the Analytical Description of Equatorial X-Ray Profiles.* J. Polymer Science (Polymer Physics Edition), v. 14, 1976, p. 921-940.
6. HUISMAN, R., and HEUVEL, H. M. *Quantitative Information from X-Ray Diffraction of Nylon-6 Yarns. II. Physical Aspects of Some Quantitative Parameters.* J. Polymer Science (Polymer Physics Edition), v. 14, 1976, p. 941-954.
7. DESPER, C. R., SOUTHERN, J. H., ULRICH, R. D., and PORTER, R. S. *Orientation and Structure of Polyethylene Crystallized Under the Orientation and Pressure Effects of a Pressure Capillary Viscometer.* J. Applied Physics, v. 41, no. 11, 1970, p. 4284-4289.

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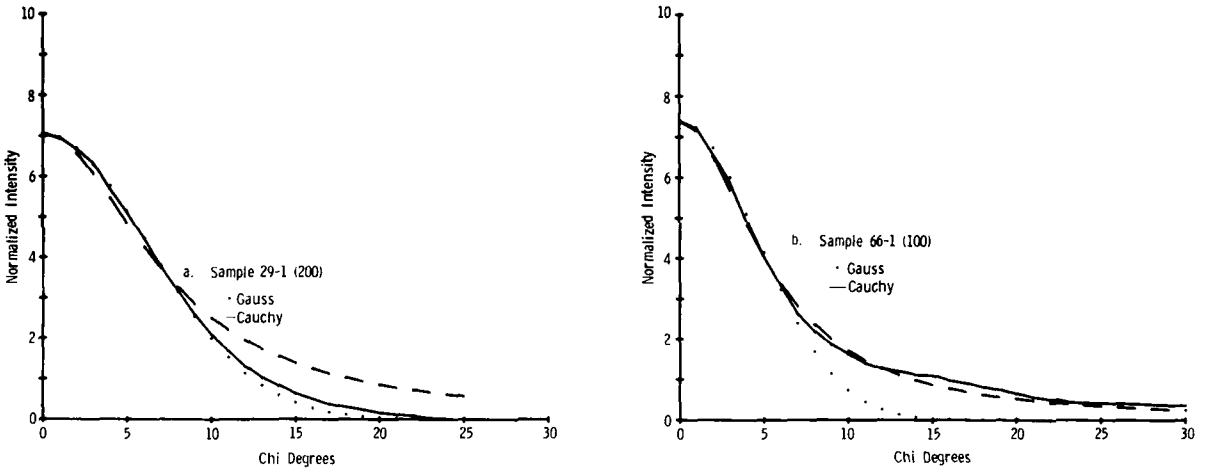

Figure 4. Orientation distribution for [a] (200) planes of sample 29-1, and [b] (100) planes of sample 66-1, compared to Gauss and Cauchy curves.

$$I = I_0 2^{-[\chi/\chi(1/2)]^2}$$

The degree of orientation of the Kevlar samples, shown in Table 6, is quite high. The f(c) values average 0.954 for the Kevlar 29 samples and 0.964 for the Kevlar 49 samples. The $\chi(1/2)$ values, which give more of a physical visualization

Table 6. CRYSTAL ORIENTATION DATA

Kevlar Samples					
	(110) Data		(200) Data		c Axis
Sample	$\chi(1/2)$, Degrees	f	$\chi(1/2)$, Degrees	f	f
29-1	7.5	-0.479	7.4	-0.479	+0.957
29-2	7.3	-0.479	7.0	-0.481	+0.959
29-3	7.2	-0.478	7.1	-0.479	+0.956
29-4	7.9	-0.473	8.0	-0.473	+0.945
29-5	7.7	-0.475	7.6	-0.476	+0.950
29-6	7.4	-0.475	7.3	-0.476	+0.950
29-7	7.2	-0.478	7.3	-0.479	+0.956
29-8	7.6	-0.476	7.5	-0.478	+0.953
29-9	7.0	-0.480	6.8	-0.481	+0.960
49-1	5.4	-0.482	4.9	-0.484	+0.966
49-2	5.4	-0.482	4.9	-0.484	+0.965
49-3	5.2	-0.480	4.9	-0.483	+0.962
Nylon 66 Sample					
	(100) Data		(010)+(110) Data		c Axis
Sample	$\chi(1/2)$, Degrees	f	$\chi(1/2)$, Degrees	f	f
66-1	5.5	-0.426	6.5	-0.450	+0.881

but have limited fundamental significance, range around 7° to 8° for the Kevlar 29 samples, around 5° to 5.5° for the Kevlar 49 samples. The differences within each fiber grade may not be significant, considering the error arising from lack of perfect parallelism in the fiber bundles.

CRYSTALLINE ORIENTATION - NYLON SAMPLE 66-1

For the nylon 66 sample, two orientation distributions were measured: that of the (100) planes diffracting at $2\theta = 20.38°$, and that of the combined (010) and (110) planes, diffracting in an unresolved peak at $2\theta = 23.18°$. The baseline technique used with the Kevlar samples (use of a straight line between $2\theta = 15°$ and 27°) was not appropriate. The actual 2θ profile at $\chi = 90°$, which showed a broad noncrystalline maximum at the $\chi = 90°$ setting, was used as the baseline instead. The experimental orientation functions f(100) and f(010+110), determined as -0.426 and -0.450, indicate a notably lower degree of orientation than was observed for the Kevlar samples, although the $\chi(1/2)$ values are comparable (see Table 6). The difference lies in the shape of the orientation distribution. As shown in Figure 4b, the distribution in nylon 66 samples resembles a Cauchy function

$$I = I_0/[1+(\chi/\chi(1/2))^2]$$

rather than a Gauss function. Because of the significantly higher tails in the Cauchy type distribution, the $\chi(1/2)$ values are misleading. The overall orientation is lower than one might expect from the $\chi(1/2)$ values.

The crystal orientation function f(c) was calculated from f(100) and f(010+110) by adapting the method previously used for polyethylene. The adaptation is based on the fact that the pattern of packing of the chains of nylon 66 in two dimensions is similar to the polyethylene pattern. A difference between the two structures is largely in the third (c axis) dimension, as indicated by Bunn and Garner,[3] and reflects the pattern of longitudinal registration required to form hydrogen bonds in the nylon 66 case. In the plane normal to the polymer chains, similar packing patterns occur, with the (100), (010), and (110) planes of the triclinic nylon 66 cell occupying positions analogous to the (200), (110), and (-110) planes of the orthorhombic polyethylene structure. To a good approximation one may use the following analog of the polyethylene equation:

$$f(c) = [(1-2\sin^2 66.25°)f(100)-f(010+110)]/\sin^2 66.25°.$$

The error in the approximation lies in the fact that the unresolved (010) and (110) pairs in nylon 66 are not related by symmetry, and thus have different scattering powers and diffraction angles, so that the experimental f(010+110) is a weighted, rather than true, average of f(010) and f(110).

An attempt was made to determine f(c) more directly using the (1,3,14) reflection of nylon 66, whose direction is within 1° of the c axis. This effort did not meet with success because the diffraction intensity was not strong enough relative to background.

SUMMARY

Of the samples submitted, the three Kevlar 49 samples show somewhat higher levels of orientation and crystal perfection than the nine Kevlar 29 samples. Within a given yarn type the effect of rupture and/or UV exposure upon a degree of orientation is insignificant; the effect upon crystal perfection is quite small. The one nylon 66 sample shows a considerably lower degree of crystal orientation than the Kevlar samples. The crystal perfection index of the nylon 66 is 72 percent, which is rather low, but its significance is ambiguous.

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Army Materials and Mechanics Research Center,
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X-RAY INVESTIGATION OF BALLISTIC YARNS -
C. Richard Desper

Technical Report AMMRC TR 79-62, December 1979, 11 pp -
illus-tables, D/A Project 1L762723AH98BD,
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Key Words

X-ray diffraction
Crystal structure
Preferred orientation

A series of ballistic polyamide yarns, comprising 12 Kevlar samples and one Nylon 66 sample, were examined by X-ray diffraction. The nine Kevlar 29 and three Kevlar 49 yarns were identified by their lattice parameters as poly(p-phenyleneterephthalamide) rather than poly(p-benzamide). The normalized equatorial scans show a somewhat greater crystallite size or perfection for the Kevlar 49 samples compared to the Kevlar 29 samples. The equatorial scan of the Nylon 66 sample yielded a Crystal Perfection Index of 72 percent. Orientation functions in the range 0.95 to 0.97 were measured for the Kevlar samples, while the corresponding value for the Nylon 66 sample was 0.881.

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